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DEVELOPMENT OF PROCESS FOR SILVER RECOVERY FROM PHOTOGRAPHIC WASTES

By  
HENRY F. HAMIL

PREPARED FOR  
NAVAL PHOTOGRAPHIC CENTER, NDW  
ANACOSTIA, BUILDING 168  
WASHINGTON, D.C. 20374  
ATTN: LT. F.R. HUDEK, PROJECT OFFICER

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HENRY F. / HAMIL

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PREPARED FOR

NAVAL PHOTOGRAPHIC CENTER, NDW  
WASHINGTON, D.C. 20374

11 May 78

APPROVED:

*G. R. Somerville*

G. R. SOMERVILLE

DIRECTOR

DEPARTMENT OF APPLIED CHEMISTRY  
AND CHEMICAL ENGINEERING



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# TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. EXPERIMENTAL	2
A. Membrane Preparation	2
B. Membrane Characterization	3
III. DISCUSSION	14
IV. CONCLUSIONS	17

## List of Tables

1. Membrane Composition	4
2. Grafting Solution Compositions	6
3. Membrane Parameters	9
4. Silver Recovery Data	12
5. Silver Removal Data	13

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## I. INTRODUCTION

This final report describes the work performed on Southwest Research Institute project 01-5030-001, Contract N00600-77-C-1103, on development of a process for silver recovery from photographic wastes and covers the period beginning 21 September 1977 and ending 21 May 1978. ✓

→ The purpose of this work was development of a silver recovery process based upon Donnan dialysis, a continuous ion exchange system in which separation of ionic species is achieved by transport across an ion exchange membrane. In Donnan dialysis, the driving force for ion transfer across the membrane <sup>is</sup> provided by a chemical concentration gradient and requires neither large hydraulic pressures, as in reverse osmosis, nor large electrical potentials, as in electrodialysis.

This program was devoted to a study of membrane characteristics and their effect on the efficiency of silver recovery from photographic wash waters.

## II. EXPERIMENTAL

### A. Membrane Preparation

The membranes prepared in this study are based upon irradiation-grafted polyethylene film. The general procedure used in membrane preparation involves backing the polyethylene film (usually 25 feet x 1.5 feet) with cheesecloth and rolling the composite onto an aluminum rod. The cheesecloth serves as a separator for the film and allows monomer solution to contact the film on both sides during grafting. Additionally, the cheesecloth prevents the film from sticking together during grafting. The rolled film is placed in a hydrometer jar which is sealed with a rubber stopper containing inlet and outlet tubes. The jar containing the film is alternately evacuated and back-flushed with nitrogen to remove oxygen. The jar is evacuated, and a degassed solution of the desired monomer in a suitable solvent is drawn into the jar to immerse the film. The filled jar is again evacuated and back-flushed with nitrogen to degas the film roll and to remove any residual oxygen. The inlet and outlet tubes are sealed and the vessel is irradiated with cobalt-60 gamma radiation to produce free radical sites in the polymer and initiate graft copolymerization. After grafting, the film is removed from the grafting vessel, separated from the cheesecloth backing, washed free of homopolymer with a suitable solvent, and air dried. The graft copolymer film is then ready for use, or for further chemical modification if required.

The base polymer used in this study was one-mil polyethylene film made from additive-free Dow 400 low density polyethylene resin. The monomers used included 2-vinylpyridine, 4-vinylpyridine, vinylbenzyl chloride, N-vinylpyrrolidone, and divinyl benzene. The latter two monomers were used to modify film hydrophilicity and to provide crosslinking, respectively. The first three monomers provided the anion exchange properties of the membranes after quaternization. Alkyl halides were utilized to quaternize the vinylpyridine-grafted films, while trialkyl amines were utilized to quaternize the vinylbenzyl chloride-grafted films.

Previous studies at Southwest Research Institute have shown that the degree of grafting (grams monomer in the product copolymer per gram of starting

polymer) is a function of the concentration of monomer in the grafting solution, which allows control of the ion exchange capacity of the product ion exchange film.

The grafting reactions were conducted at ambient temperature with an irradiation dose rate of 12,000 rads per hour. Run duration was 68 hours. The total applied radiation dose was 0.816 megarad.

A list of graft copolymers produced for this study, along with the reagent used in the quaternization reaction, is given in Table 1. A list of the grafting solutions, showing monomer concentration and solvent, used in preparation of the graft copolymers is presented in Table 2.

#### B. Membrane Characterization

Prior to evaluation in the Donnan dialysis cell, the ion exchange capacity and equilibrium water content of each membrane were determined.

The equilibrium water content, defined as grams of water absorbed per gram of dry polymer, was determined by placing a portion of the membrane in deionized water for 24 hours. The membrane was then removed from the water, blotted free of surface water with absorbent toweling, and placed in a tared vial. After the wet weight of the membrane was determined, the membrane and vial were dried to constant weight at 110°C. The dry weight of the membrane was determined, and the equilibrium water content was calculated from the wet and dry membrane weights.

The ion exchange capacity, defined as milliequivalents per gram of dry membrane, was determined as follows. A portion of the membrane was converted to the chloride form by repeated treatments with 10 volume percent hydrochloric acid. The membrane was then dried. Initially, a determination of weak base ion exchange capacity was made, where the weak base capacity is measured relative to dilute ammonium hydroxide. A weighed portion of the membrane in the chloride form is placed into a measured volume of one percent ammonium hydroxide and agitated for at least six hours. The chloride content of the ammonical solution is then determined titrimetrically by the Volhard method. The weak base ion exchange capacity is calculated from the



TABLE 1. MEMBRANE COMPOSITION

<u>Membrane No.</u>	<u>Graft Copolymer</u>	<u>Quaternizing Reagent</u>
N1Q	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N2Q	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N3Q	poly [ethylene-g-(4-vinyl pyridine-co-N-vinyl pyrrolidone)]	Methyl Iodide
N4Q1	poly (ethylene-g-2-vinyl pyridine)	Methyl Iodide
N4Q2	poly(ethylene-g-2-vinyl pyridine)	2-Chloroethanol
N5Q1	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N5Q2	poly(ethylene-g-2-vinyl pyridine)	2-chloroethanol
N6Q1	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N6Q2	poly(ethylene-g-4-vinyl pyridine)	2-chloroethanol
N7Q1	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N7Q2	poly(ethylene-g-4-vinyl pyridine)	2-chloroethanol
N8Q1	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N8Q2	poly(ethylene-g-vinylbenzyl chloride)	Triethanolamine
N9Q1	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N9Q2	poly(ethylene-g-vinylbenzyl chloride)	Triethanolamine
N4Q3	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N5Q3	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N6Q3	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N7Q3	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N10Q1	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N10XQ1	poly [ethylene-g-(4-vinyl pyridine-co-divinylbenzene)]	Methyl Iodide
N10Q3	poly(ethylene-g-4-vinyl pyridine)	Dodecyl bromide
N11Q1	poly(ethylene-g-4-vinyl pyridine)	Methyl Iodide
N11XQ1	poly [ethylene-g(4-vinyl pyridine-co-divinyl pyridine)]	Methyl Iodide
N11Q3	poly(ethylene-g-4-vinyl pyridine)	Dodecyl bromide
N12Q1	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N13Q1	poly(ethylene-g-2-vinyl pyridine)	Methyl Iodide
N14Q1	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N14XQ2	poly [ethylene-g-(vinyl benzyl chloride-co-divinylbenzene)]	Trimethylamine



Table 1. Membrane Composition (cont'd)

<u>Membrane No.</u>	<u>Graft Copolymer</u>	<u>Quaternizing Reagent</u>
N14Q4	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N15Q1	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N15XQ2	poly [ethylene-g-(vinylbenzyl chloride-co-divinyl benzene)]	Trimethylamine
N15Q4	poly(ethylene-g-vinylbenzyl chloride)	Tri-N-butylamine
N16Q1	poly(ethylene-g-vinylbenzyl chloride)	Trimethylamine
N16XQ2	poly [ethylene-g-(vinylbenzyl chloride-co-divinyl benzene)]	Trimethylamine

TABLE 2. GRAFTING SOLUTION COMPOSITIONS

<u>Base Copolymer No.</u>	<u>Grafting Solution</u>
N-1	2-vinylpyridine, 25 wt.% in methanol
N-2	4-vinylpyridine, 25 wt.% in methanol
N-3	4-vinylpyridine, 25 wt.% and N-vinylpyrrolidone, 15 wt.% in methanol
N-4	2-vinylpyridine, 10 wt.% in methanol
N-5	2-vinylpyridine, 20 wt.% in methanol
N-6	4-vinylpyridine, 10 wt.% in methanol
N-7	4-vinylpyridine, 20 wt.% in methanol
N-8	Vinylbenzyl chloride, 10 wt.% in benzene
N-9	Vinylbenzyl chloride, 20 wt.% in benzene
N-10	4-vinylpyridine, 25 wt.% in methanol
N-10X	Divinylbenzene, 5 wt.% in methanol containing 5 wt.% benzene
N-11	4-vinylpyridine, 35 wt.% in methanol
N-11X	Divinylbenzene, 5 wt.% in methanol containing 5 wt.% benzene.
N-12	2-vinylpyridine, 25 wt.% in methanol
N-13	2-vinylpyridine, 35 wt.% in methanol
N-14	Vinylbenzyl chloride, 40 wt.% in benzene
N-14X	Divinylbenzene, 5 wt.% in methanol containing 5 wt.% benzene.
N-15	Vinylbenzyl chloride, 30 wt.% in benzene.
N-15X	Divinylbenzene, 5 wt.% in methanol containing 5 wt.% benzene.
N-16	Vinylbenzyl chloride, 50 wt.% in benzene.
N-16X	Divinylbenzene, 5 wt.% in methanol containing 5 wt.% benzene.

milliequivalents of chloride ion liberated and the weight of the membrane used.

The strong base ion exchange capacity is then determined by taking the membrane sample from the weak base determination, rinsing it with deionized water, and placing it in a measured volume of neutral four weight percent  $\text{NaNO}_3$  solution. After six hours agitation, the liberated chloride is determined by the Volhard method. The strong base ion exchange capacity is then calculated based upon chloride ion liberated and membrane weights. The sum of the weak and strong ion exchange capacities gives the total ion exchange capacity of the membrane.

It should be noted that even though quaternized vinylpyridine-grafted membranes are quaternary ammonium compounds, in the above ion exchange determinations, they appear to be weak base ion exchange materials, i.e., they are less basic than the dilute ammonium hydroxide used in the determination. Presumably, this is due to the fact that the nitrogen atom in these materials is located in an aromatic ring, which results in stabilization of the positive charge on nitrogen by resonance effects. This results in a decrease in basicity.

The membranes were evaluated for osmotic water transport and silver transport by Donnan dialysis in a single membrane dialysis cell operated in a recycle batch mode. The test cell consisted of end plates, polypropylene mesh flow spacers with gasketed edges, and the test membrane. Active membrane area was  $180 \text{ cm}^2$ , the flow channels on either side of the membrane were 0.04 cm thick, and the volume of each cell half was  $15 \text{ cm}^3$ .

The osmotic water transport was determined by installing a membrane in the test cell, running a leak check, and then determining the volume of water transferred across the membrane when deionized water was fed to one cell half and 0.392 N  $\text{NaNO}_3$  solution was fed to the other cell half. Volume measurements of the salt solution were made periodically, and a plot of incremental volume change versus time was made. The slope of the plot gives the rate of osmotic water flow across the membrane. This value is divided by the active membrane area to give the osmotic water flow rate in units of milliliters water per minute per  $\text{cm}^2$  membrane under the conditions of the experiment.



The total ion exchange capacities, the osmotic water transport rates, and the equilibrium water contents for the membranes used in this study are given in Table 3.

The silver transport for the membranes was determined by using six liters of synthetic photographic wash water as feed to one side of the Donnan dialysis cell. A stripping solution consisting of 0.3 liter of the above feed containing 10 grams of sodium nitrate (0.392 moles/liter) was fed to the other side of the cell. These solutions were recirculated through the cell for eight hours. Prior to the run, and at one-hour intervals during the run, samples of feed and stripping solution were taken and analyzed for silver content by atomic absorption spectroscopy. The amount of silver removed from the feed was calculated from the silver analysis and the feed solution volume. Similarly, the amount of silver appearing in the stripping solution was calculated. The percent silver removal was calculated based upon the initial and final silver content of the feed solution. The percent silver accountability was calculated using the weight of silver removed from the feed solution and the weight of silver appearing in the stripping solution. A concentration factor was calculated by dividing the initial concentration of silver in the feed into the final silver concentration in the stripping solution.

The rate of silver removal from the feed solution in a properly operating dialysis system obeys a first order rate law. The rate constant for silver removal in a Donnan dialysis system can be calculated according to the following equation:

$$k = - \left( \frac{1}{t} \ln \frac{C_0}{C_t} \right)$$

Where  $k$  is the first order rate constant,  $C_0$  is the initial concentration,  $C_t$  is the concentration at any time  $t$ , and  $t$  is the elapsed time. Since all membrane evaluations were conducted under the same experimental conditions, calculations of the silver removal rate constant allows a quantitative comparison of membrane performance. Additionally, calculation of the rate constant on an hour by hour basis for each run gives an indication of membrane performance with time.

TABLE 3. MEMBRANE PARAMETERS

Membrane No.	Total Ion (1) Exchange Capacity meq/g	Osmotic Water (2) Transport Rate $\text{ml/cm}^2 \text{ min} \times 10^4$	Equilibrium Water Content g/g
N1Q	0.85	0.15	0.65
N2Q	1.00	1.39	0.57
N3Q	1.55	1.28	1.95
N4Q1	0.47	--*	0.22
N4Q2	0.20	--*	0.29
N5Q1	0.66	0.25	0.53
N5Q2	0.50	0.19	0.21
N6Q1	1.31	0.17	0.48
N6Q2	1.01	0.85	0.24
N7Q1	1.75	1.59	0.56
N7Q2	0.39	--*	0.50
N8Q1	0.31	--*	0.22
N8Q2	0.17	--*	0.08
N9Q1	0.88	0.35	0.48
N9Q2	0.40	--*	0.10
N4Q3	0.53	--*	0.25
N5Q3	0.66	--*	0.26
N6Q3	0.89	0.36	0.27
N7Q3	1.46	--*	0.48
N10Q1	1.19	5.0	1.50
N10XQ1	1.17	5.2	1.45
N10Q3	0.29	2.04	0.55
N11Q1	1.41	7.03	3.20
N11XQ1	1.37	5.77	1.86
N11Q3	0.40	3.02	0.20
N12Q1	0.30	0.28	0.27
N13Q1	0.26	0.37	0.35

TABLE 3. Membrane Parameters (cont'd)

N14Q1	2.47	7.69	0.94
N14XQ2	2.12	9.15	2.55
N14Q4	1.31	0.35	0.22
N15Q1	2.90	8.43	1.26
N15XQ2	2.28	7.13	1.05
N15Q4	1.24	0.13	0.35
N16Q1	2.97	9.15	1.29
N16XQ2	2.53	--*	1.19

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(1) The ion exchange capacities are reported as the sum of the weak base and strong base capacities. Quaternized vinylpyridines behave as weak base ion exchangers in the analytical procedures used, even though they are quaternary ammonium bases.

(2) Determined using deionized  $H_2O$ : 0.392 N  $NaNO_3$  across the membrane.

\* membranes failed leak test. No determination made.



The run parameters, described above, for the membranes used in this study are given in Tables 4 and 5.

TABLE 4. SILVER RECOVERY DATA

<u>Membrane No.</u>	<u>Silver Removal, %</u>	<u>Silver Accountability, %</u>	<u>Concentration Factor</u>	<u>Silver Removal Rate Constant, hr<sup>-1</sup></u>
N1Q	77.1	87.2	16.2	-0.19
N2Q	95.3	98.4	21.7	-0.58
N3Q	95.5	94.1	21.5	-0.65
N5Q1	0	--	--	0
N5Q2	0	--	--	0
N6Q1	0	--	--	0
N6Q2	46.7	102.4	11.36	-0.08
N6Q3	63.4	90.2	13.0	-0.16
N7Q1	99.5	95.5	16.4	-0.66
N9Q1	36.0	94.7	9.1	variable
N12Q1	0	--	--	0
N13Q1	40.9	102.7	10.9	-0.09
N10Q3	0	--	--	0
N11Q3	0	--	--	0
N14Q4	0	--	--	0
N15Q4	0	--	--	0

TABLE 5. SILVER REMOVAL DATA

Membrane No.	M A X I M U M				F I N A L				Initial Silver Removal Rate Constant hr-1
	Silver Removal, %	Silver Accountability, %	Concentration Factor	Silver Removal, %	Silver Accountability, %	Concentration Factor	Concentration Factor		
N10Q1	69.1	94.2	14.8	60.9	90.5	12.8			-0.47
N11Q1	72.0	82.1	14.9	55.2	49.9	7.2			-0.57
N14Q1	80.3	95.4	16.3	71	88.9	13.6			-0.64
N15Q1	77.9	96.8	16.7	70.8	99.0	14.1			-0.62
N16Q1	73.8	93.0	15.4	55.5	85.3	9.8			-0.61
N10XQ1	67.5	97.4	15.5	59.3	91.8	13.6			-0.37
N11XQ1	59.4	92.5	13.2	35.4	84.3	7.9			-0.48
N14XQ2	78.8	104.4	19.9	63.5	103.4	15.3			-0.67
N15XQ2	79.1	100.1	16.8	63.6	97.8	13.1			-0.69



### III. DISCUSSION

The membranes prepared for this study cover a wide range of properties, as shown in Table 3. Thirty-five membranes were prepared, with a range of ion exchange capacities from 0.17 to 2.97 milliequivalents per gram, equilibrium water contents from 0.08 to 3.20 grams per gram, and osmotic water transport rates from  $0.13 \times 10^{-4}$  to  $9.15 \times 10^{-4}$  ml per minute per  $\text{cm}^2$ . Twenty-five of the membranes were evaluated in Donnan dialysis. The remaining ten were not evaluated in Donnan dialysis due to leaks. During membrane preparation, some of the membranes were found to be somewhat brittle after grafting and before quaternization. Cracks developed during handling which led to leaks in the product membranes.

The twenty-five membranes which were evaluated were divided into two categories: membranes which behaved normally in that they transported silver over the entire evaluation period with no significant change in the silver removal rate constant, and membranes which displayed an anomalous behavior in that they initially transported silver normally but the value of the rate constant decreased with time until a reversal of silver flow was observed.

The parameters of the membranes in the first category are presented in Table 4, and those of the latter category are presented in Table 5.

Those membranes reported as normal include three with excellent transport properties ( >95 percent removal ) two with fair properties (63-77 percent removal), three with poor properties (36-46% removal), and eight with essentially zero transport properties. The three membranes with the best properties (N2Q, N3Q, N7Q1) had ion exchange capacities of 1.0-1.75 meq/g and osmotic water transport rates of  $1.28-1.59 \times 10^{-4}$  ml per minute per  $\text{cm}^2$ . The two fair membranes (N1Q and N6Q3) had ion exchange capacities of 0.85 and 0.89 meq/g, respectively, and osmotic water transport rates of  $0.15$  and  $0.36 \times 10^{-4}$  ml per minute per  $\text{cm}^2$ . The three poor membranes (N6Q2, N9Q1, N13Q1) had respective ion exchange capacities of 1.01, 0.88, and 0.26 meq/g and respective osmotic water transport rates of 0.85, 0.35 and  $0.37 \times 10^{-4}$  ml per minute per  $\text{cm}^2$ . The membranes with essentially zero

transport had combinations of the above two parameters in which one or both parameters was on the low side. While the effect of the interaction of these parameters on silver transport cannot be quantitated, it appears that there may be optimal levels of ion exchange capacity and osmotic water transport rate which in combination produce acceptable membranes.

The parameters of the membranes reported as anomalous all show the same characteristic behavior. Silver removal initially occurred at a reasonable rate, with rate constants in the range of  $-0.37 \text{ hr}^{-1}$  to  $-0.69 \text{ hr}^{-1}$ , as compared to rate constants of  $-0.58 \text{ hr}^{-1}$  to  $-0.66 \text{ hr}^{-1}$  for the best normal membranes. However, the rate constant for the anomalous membranes decreased with time as the run proceeded. At about the mid point of the runs, maximum silver removal was obtained, being in the range of about 60 to 80 percent. At this point, the flow of silver across the membrane reversed, and the concentration of silver in the stripping solution decreased. The final silver removal obtained was in the range of about 35 to 70 percent. The reason for this loss of permselectivity is not completely understood. However, examination of the other membrane parameters shows that these membranes all had quite high ion exchange capacities (1.17-2.97 meq/g) and quite high osmotic water transport rates ( $5.0 - 9.15 \times 10^{-4} \text{ ml/min cm}^2$ ). Since higher ion exchange capacity should enhance the silver transfer rate, the high osmotic water transport rates may indicate the problem. This latter parameter is a function of membrane hydrophilicity. As the membranes become more hydrophilic, they imbibe more water and become quite swollen. This effectively increases the wet volume of the membrane and may lead to a decrease in the effective charge density of the fixed positive charge per unit volume in the swollen membrane. Such a decrease in fixed charge density would decrease the ionic repulsion for mobile cations in the solution and would result in a decreased ion selectivity in the membrane.

It should be pointed out that the anomalous behavior of these membranes is a reversible phenomenon. Replicate runs were made with various membranes without removing the membrane from the cell. Fresh feed and stripping solutions were used. The same initial silver removal rate constant was obtained, within experimental error, on these runs. The same pattern of

decreasing silver transfer and ultimate reversal of silver flow were obtained.



#### IV. CONCLUSIONS

✓  
The role of intrinsic membrane parameters on the transport of ions in Donnan dialysis appears more complex than originally postulated.

The available data indicate that there are optimal values for ion exchange capacity and membrane hydrophilicity. If ion exchange capacity or hydrophilicity are too low, no ion transport occurs. Increasing ion exchange capacity appears to increase the ion transport rates, but the attendant increase in membrane hydrophilicity leads to a loss of ion permselectivity. It thus appears that a balance in ion exchange capacity and hydrophilicity must be achieved to provide an acceptable membrane.

While these conclusions should be considered tentative, it appears that this is the best explanation of the results obtained in this study based upon the available data.

It is recommended that further membrane study be conducted prior to any work toward developing a prototype Donnan dialysis system for silver recovery.